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PATENT ABSTRACTS OF JAPAN

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(54) HARD FILM-COATED MEMBER

(57)Abstract:
PROBLEM TO BE SOLVED: To improve the wear resistance, oxidation resistance and lubricity in a hard film-coated member to which a hard film is applied.
SOLUTION: In this hard film-coated member to which a hard film is applied, the hard film is coated with a film containing at least Al, Ti, Cr, N and O by one or more layers.

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CLAIMS

[Claim(s)]
[Claim 1] It is the hard-anodic-oxidation-coatings covering member characterized by covering the coat in which this hard anodic oxidation coatings contains aluminum, Ti, Cr, N, and O at least in the hard-anodic-oxidation-coatings covering member which covered the hard anodic oxidation coatings one layer or more than two-layer.
[Claim 2] composition (NwO(AlaTibCrc)1-w) of the layer which constitutes this hard anodic oxidation coatings in a hard-anodic-oxidation-coatings covering member according to claim 1 -- however, 30<=a<=70, 30<=b<=70, 0.5<=c<=20, a+b+c=100, 0.7<=w<=0.99, and the hard-anodic-oxidation-coatings covering member that comes out and is characterized by a certain thing
[Claim 3] It is the hard-anodic-oxidation-coatings covering member characterized by this number of layers being 3-1000 layers in a hard-anodic-oxidation-coatings covering member according to claim 1 to 2.
[Claim 4] It is the hard-anodic-oxidation-coatings covering member characterized by the thickness of this layer being 5nm - 2000nm in a hard-anodic-oxidation-coatings covering member according to claim 1 to 3.
[Claim 5] The hard-anodic-oxidation-coatings covering member characterized by constituting a hard anodic oxidation coatings from A layers with few oxygen contents, and a B horizon with many oxygen contents in a hard-anodic-oxidation-coatings covering member according to claim 1 to 3.
[Claim 6] The oxygen content of one to 10 atom % and this B horizon is a hard-anodic-oxidation-coatings covering member characterized by the oxygen content of this A layer being ten to 30 atom % in a hard-anodic-oxidation-coatings covering member according to claim 5.
[Claim 7] The hard-anodic-oxidation-coatings covering member characterized by covering a part or the whole of this A layer and/or a B horizon, and an oxygen content having inclination composition in a hard-anodic-oxidation-coatings covering member according to claim 5 to 6.
[Claim 8] The hard-anodic-oxidation-coatings covering member characterized by the crystal structure of this hard anodic oxidation coatings being NaCl type in a hard-anodic-oxidation-coatings covering member according to claim 1 to 7.
[Claim 9] The hard-anodic-oxidation-coatings covering member characterized by the value of I (200)/I (111) being one or more in a hard-anodic-oxidation-coatings covering member according to claim 1 to 8 when the diffraction intensity of I (200) and a ** (111) side is set to I (111) for the diffraction intensity of the field in X ****** (200) of this hard anodic oxidation coatings.
[Claim 10] The hard-anodic-oxidation-coatings covering member to which this hard-anodic-oxidation-coatings crystal gestalt is characterized by the detailed columnar crystal or the amorphous thing in a hard-anodic-oxidation-coatings covering member according to claim 1 to 9.
[Claim 11] The hard-anodic-oxidation-coatings covering member to which particle size of this detailed columnar crystal is characterized by being 250nm or less in the position of 1000nm - 1500nm from the interface of a hard anodic oxidation coatings and a base in a hard-anodic-oxidation-coatings covering member according to claim 1 to 10.
[Claim 12] The hard-anodic-oxidation-coatings clothing member characterized by the compressive stress which remains to this hard anodic oxidation coatings being 3.5 or less GPas in a hard-anodic-oxidation-coatings covering member according to claim 1 to 11.

[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[The technical field to which invention belongs] this invention relates to the wear-resistant member which has high abrasion resistance and oxidation resistance while it is excellent in solid-lubrication nature.
[0002]
[Description of the Prior Art] In order to give abrasion resistance, oxidation resistance, and lubricity in a tool metallurgy type and a machine part, it is general to cover various hard anodic oxidation coatingss. Although typical TiN and TiCN coat are excellent in abrasion resistance, they have a problem in oxidation resistance. Moreover, lubricity is low although TiAlN system coat proposed by JP,62-56565,A etc. is excellent in abrasion resistance and oxidation resistance. Although CrN and CrCN system coat are excellent, a coat degree of hardness is low and they are inferior to abrasion resistance in lubricity. Thus, the present condition is the result of the conventional coat being inferior in abrasion resistance, oxidation resistance, and lubricative either, and having a certain trouble in each intended use. Although the proposal which covers MoS system coat which is excellent in lubricity on a hard-anodic-oxidation-coatings front face is made by JP,5-239618,A etc. in order to give lubricity, it has come moreover,] to obtain result with it. [bad adhesion and] [sufficient] Thus, although the proposal which covers CrN system coat in the upper layer of TiAlN system coat is also made as shown in JP,11-156992,A in addition to MoS system in order for there to be a certain trouble and to solve it, since the whole thickness has a certain amount of limit, it is not satisfied of the thickness of TiAlN coat with the conventional coat in respect of not being enough and abrasion resistance.
[0003]
[Problem(s) to be Solved by the Invention] this invention aims at raising these properties, without degrading abrasion resistance, oxidation resistance, and lubricative all.
[0004]
[Means for Solving the Problem] In the hard-anodic-oxidation-coatings covering member with which this invention covered the hard anodic oxidation coatings in order to solve the above-mentioned technical probrem, this hard anodic oxidation coatings covers the coat which contains aluminum, Ti, Cr, N, and O at least one layer or more than two-layer. Moreover, composition of each class which constitutes a hard anodic oxidation coatings as an embodiment superior to this invention's (AlaTibCrc) ** (NwO1-w) however, 30<=a<=70, 30<=b<=70, 0.5<=c<=20, a+b+c=100, 0.7<=w<=0.99, and a sushi -- The thickness of each class which the number of layers makes 3-1000 layers sets to 5nm - 2000nm. Should constitute the hard anodic oxidation coatings from A layers with few oxygen contents, and a B horizon with many oxygen contents. The oxygen content of A layers makes the oxygen content of one to 10 atom %, and a B horizon ten to 30 atom %. Use as NaCl type the crystal structure of a hard anodic oxidation coatings which covers a part of each class or whole each class, and considers an oxygen content as inclination composition. The diffraction intensity of the field in X ****** (200) of a hard anodic oxidation coatings I (200), When the diffraction intensity of a ** (111) side is set to I (111), make the value of I (200)/I (111) or more into one. Setting to 3.5 or less Gpas compressive stress to which the particle size of a detailed columnar crystal which makes the hard-anodic-oxidation-coatings crystal gestalt a detailed columnar crystal or the amorphous gestalt remains to a hard anodic oxidation coatings and the hard anodic oxidation coatings set to 250nm or less in the position of 1000nm - 1500nm from the interface of a base etc. is mentioned. this invention uses Ti, aluminum, and Cr and N as an indispensable component, and adopts the hard anodic oxidation coatings which added oxygen to this. Although Ti and aluminum contribute as a wear-resistant component with natural and it contributes as a component which Cr gives lubricity, just this is not enough and the result which gives oxidation resistance and lubricity much more is brought by adding oxygen to this.
[0005]
[Function] Especially about the effect which adds Cr to this on the basis of TiAlN, the further oxidation-resistant enhancement is mentioned in the first place in a cutting tool. It is a well-known fact that in TiAlN aluminum is spread on a front face inside a coat in connection with oxidization, suppress permeation from the exterior of oxygen by forming the oxide of aluminum, and oxidation resistance improves. However, since the oxide of very porous Ti is formed directly under the oxide of aluminum in this case, when especially the impact of a cutting tool etc. is applied, it is difficult [it] for the oxide of aluminum to be easily omitted, and to continue and demonstrate the effect. It is the same also at the case of metal mold etc. The oxide of porous Ti formed directly under [oxide] aluminum of addition of Cr turned into the oxide of TiCr, and this oxide became clear [forming a very precise layer]. Therefore, adhesion will be maintained and result oxidation resistance of the oxide of aluminum formed in the maximum cortex will improve.
[0006] The second effect of Cr addition is giving the outstanding lubricity which the Cr's itself has to TiAlN coat. Although coefficient of friction with the steel of TiAlN is 0.7-0.8, it is improved by 0.3-0.6 in connection with Cr addition. It depends for this coefficient of friction on the addition of Cr. However, since a coat degree of hardness will fall and abrasion resistance will become bad if there are too many additions of Cr, it is desirable to set an upper limit to an addition.
[0007] Although the effect that only Cr addition makes the lubricity of TiAlN system coat and oxidation resistance improve as mentioned above but is checked, just Cr is not enough and still much more improvement accepts by adding oxygen to this. About the addition effect of oxygen, the oxidation-resistant enhancement in fast and the lubricative enhancement in fast are mentioned in the first place. While the crystal of a coat makes it detailed and the coat itself becomes precise in connection with adding oxygen inside a coat to the oxidation-resistant enhancement in fast, when the grain boundary also becomes precise, it is considered to be the ground that the oxygen diffusion rate to permeation

of the oxygen from the exterior is suppressed remarkably. Although not fully solved about lubricity, it is considered to be the ground that the compatibility with steel falls by the oxygenation.
[0008] The residual compression stress of the second coat of the oxygenation effect declines, and the wear-resistant enhancement resulting from the adhesion of a coat improving is mentioned. especially -- the bottom of a deep-cuts condition, and forging -- metal mold -- in a field, the adhesion of a coat serves as a very important element It will become a sudden life if big sublation occurs while it is in the inclination that wear advances by minute sublation of a coat. The intensity is improved more than 100N by adding oxygen to this to the sublation critical load by the scratch test of the coat of TiAlCrN system being 60-80N.
[0009] However, although the abrasion resistance which originates in above-mentioned oxidation resistance, lubricity, and the enhancement in adhesion in connection with the increase in an addition of oxygen improves, it brings the result in which the coat degree of hardness itself softens on the other hand, and abb ******** wear nature deteriorates. Therefore, it is important that the optimum composition layer for contributing to oxidation resistance and lubricity and the optimum composition layer contributed to abb ******** wear are multilayered, it is more desirable, and the result by which both advantage is composite-ized by multilayering is brought.
[0010] Next, the ground which limited the numeric value is explained. Since the oxidation resistance of a coat will deteriorate if it is under 30 atom %, AlN which has an hcp structure will be formed into a coat and a coat intensity will deteriorate if 70 atom % is exceeded, aluminum is not desirable. Since the abrasion resistance of a coat will deteriorate if it is under 30 atom %, and the oxidation resistance of a coat will deteriorate if 70 atom % is exceeded, Ti is not desirable. If porous Ti oxide is formed, and Cr does not contribute to oxidation-resistant enhancement but 20 atom % is exceeded, the degree of hardness of a coat softens, and its abrasion resistance deteriorates and is not desirable at under 0.5 atom %. Oxygen is not desirable in order to cause malacia of a coat degree of hardness, if it does not contribute to the enhancement in oxidation resistance, lubricity, and adhesion but 30 atom % is exceeded under by 1 atom % to nitrogen.
[0011] If one of faults will become remarkable, the compound effect will not accept, as it mentioned above, although each effect was demonstrated, if the multilayer number of coats is less than three layers, but 1000 layers are exceeded, since there is an inclination which residual stress increases and spoils the adhesion of a coat while it similarly stops the compound effect accepting thinly, the thickness of each class is not desirable. The same is said of the thickness of each class, and if the effect of the advantage which each class has in less than 5nm fades and 2000nm is exceeded, the actuality of covering of about at most three layers is lost, and it is not desirable.
[0012] As mentioned above, a hypoxia coat has few degree-of-hardness falls, the purpose which makes a multilayer a hypoxia inclusion layer and a hyperoxia inclusion layer contributes to abb-proof ******** wear, although a coat degree of hardness tends to fall, both effect is composite-ized by an anti-oxidation and the effect contributed to lubricity being large, and covering these to a multilayer, and a hyperoxia inclusion coat brings a desirable result. In a hypoxia inclusion layer, since abb-proof ******** wear nature will deteriorate if the adhesion with a hyperoxia inclusion layer deteriorates and an oxygen content exceeds 10 atom % under by 1 atom %, it is not desirable. On the other hand, if the oxygen content of contribution is thin to oxidation resistance and lubricative enhancement at under 10 atom % and 30 atom % is exceeded in a hyperoxia inclusion coat, a degree of hardness will soften remarkably, abrasion resistance is degraded, and it is not desirable.
[0013] Although a problem does not have a multilayer with simple this hypoxia inclusion coat and hyperoxia inclusion coat in any way, either, the adhesion of each class brings the result which improves further by making an oxygen content inclination-ize in each class, lessening change of an oxygen content in an interface as much as possible, or making an oxygen content continuity-ize like a sign curve.
[0014] In the crystal gestalt, since NaCl type has many sliding surfaces, an about 3000-HV upper limit exists in the coat degree of hardness in an elevated temperature, and the life which there was little occurrence of the micro crack in a coat, and was stabilized when it excelled in opposite side toughness although a raise beyond this in a degree of hardness was difficult, and occurrence of a chipping and impact were applied is attained.
[0015] If the crystal orientation of a coat forms membranes by the low energy comparatively depending on covering conditions (200), orientation of it will be strongly carried out to a field, and when membranes are formed by the high energy (111), it has the inclination which carries out orientation to a field. Although, as for the membrane formation speed of a coat, the case where membranes were formed by the low energy became late, a density of film improves and bringing the result excellent in oxidation resistance and abrasion resistance was checked. Therefore (200), when a field diffraction intensity is stronger than a field (111) diffraction intensity, further excellent oxidation resistance and abrasion resistance are demonstrated, and it is thought that it is more desirable. In addition, in lubricity, crystal orientation was not what does big influence.
[0016] The diameter of crystal grain of a coat is determined in the fracture surface SEM, sets up ** parallel to a base in the position of 1000nm - 1500nm from a base front face, and specifies it from the number of the grain boundaries which cross this. In this case, if the diameter of coat crystal grain exceeds and makes 250nm big and rough, abrasion resistance and a coat intensity deteriorate and are not desirable. Although the shape of an amorphous substance is not amorphous in fact here, the case where the clear grain boundary is not observed on fracture surface observation is said. In such a case, especially oxidation-resistant remarkable enhancement is checked.
[0017] Since adhesion will deteriorate if 3.5Gpa is exceeded although it is mainly dependent on covering conditions, the compressive stress which remains to a coat is not desirable. In addition, even if the coats of this invention are any of an arc ion-plating method, a sputtering method, an electron gun vaporization method, and a plasma assistant CVD method, the inclination may be the same and may be the combination of these all directions formula.
[0018]
[Example] Next, the desirable example of this invention is explained with the example of a comparison. The coat of the example of this invention and the example of a comparison was created with the arc ion-plating method. Composition of AlTiCr was adjusted by adjusting metal composition of the cathode target which is an evaporation source. The oxygen content was multilayered by change of gas while it was adjusted by adjusting the mixing ratio using the mixed gas of nitrogen and oxygen. The crystal orientation top was fundamentally adjusted on covering conditions, the orientation (200) coat set 70V reaction pressure to 1Pa for the bias voltage which ****s covering conditions to a base, and the orientation (111) coat created bias voltage by setting 200V reaction pressure to 0.5Pa. In addition to this, I (200) / I (111) ratio is somewhat dependent also on coat composition and an oxygen content.
[0019]
[Table 1]

	試料番号	A層	B層	層数
本発明例	1	50Al40Ti10Cr-5095N	50Al40Ti10Cr-25075N	20
	2	55Al35Ti10Cr-5095N	55Al35Ti10Cr-25075N	20
	3	35Al55Ti10Cr-5095N	35Al55Ti10Cr-25075N	20
	4	65Al32Ti3Cr-5095N	65Al32Ti3Cr-25075N	20
	5	33Al64Ti3Cr-5095N	33Al64Ti3Cr-25075N	20
	6	40Al35Ti25Cr-5095N	40Al35Ti25Cr-25075N	20
	7	50Al40Ti10Cr-2095N	50Al40Ti10Cr-25075N	20
	8	50Al40Ti10Cr-5095N	50Al40Ti10Cr-13087N	20
	9	50Al40Ti10Cr-5095N	50Al40Ti10Cr-25075N	4
	10	〃	〃	100
	11	〃	〃	500
	12	〃	〃	900
	13	50Al40Ti10Cr-(10-1-10)0(80-99-90)N傾斜	50Al40Ti10Cr-(10-25-10)0(90-75-80)N傾斜	20
	14	40Al35Ti25Cr-5095N	65Al32Ti3Cr-15085N	20
	15	33Al64Ti3Cr-7093N	40Al35Ti25Cr-15085N	20
比較例	16	TiN	-	1
	17	Ti-50N50C	-	1
	18	50Al50TiN	-	1
	19	TiN(500nm)	50Al50TiN(2500nm)	2
	20	65Al35TiN	-	1
	21	65Al35TiN	100nmMoS2	2
	22	65Al35TiN(2000nm)	CrN(1000nm)	2
	23	50Al50Ti-70N30C	-	1
	24	TiN	50Al50TiN	20
	25	50Al40Ti10CrN	-	1
	26	50Al40Ti10Cr-5095N	-	1
	27	60Al40Ti10Cr-25075N	-	1
	28	50Al40Ti10Cr-5095N	50Al40Ti10Cr-50050N	20
	29	〃	〃	1500

[0020] The detail of the example of this invention and the example of a comparison is shown in Table 1. In addition, the thickness of a coat was altogether unified by 3000nm - 3200nm in the example of this invention, and the example of a comparison this time.

[0021]

[Table 2]

	試料番号	硬度(HV)	I(200)/I(111)	酸化増量 mg/min	摩擦係数	応力 Gpa	粒径 nm
本発明例	1	2650	5.23	1.87	0.35	1.26	160
	2	2700	4.22	1.11	0.34	1.35	148
	3	2570	7.11	3.66	0.38	1.05	220
	4	2750	3.25	1.43	0.33	2.04	158
	5	2480	4.33	3.10	0.32	2.22	210
	6	2500	2.69	0.98	0.38	1.69	非晶質
	7	2670	2.58	1.98	0.39	2.46	201
	8	2710	2.77	2.31	0.40	2.41	175
	9	2630	6.14	1.78	0.35	1.02	210
	10	2690	4.28	1.55	0.34	1.96	143
	11	2750	3.44	1.43	0.33	2.31	121
	12	2600	2.49	1.40	0.33	2.93	98
	13	2680	5.03	1.79	0.32	1.76	158
	14	2600	4.34	1.23	0.38	1.89	125
	15	2690	3.69	1.49	0.32	1.97	129
比較例	16	2160	0.25	65.28	0.67	1.67	350
	17	2980	0.22	98.13	0.29	4.02	258
	18	2700	2.43	11.67	0.66	2.69	341
	19	2700	0.58	12.55	0.65	3.97	289
	20	2750	3.22	8.05	0.69	2.11	253
	21	2720	3.22	22.23	0.11	2.78	331
	22	2240	3.22	15.67	0.29	2.88	332
	23	3010	2.58	25.44	0.65	3.97	247
	24	2460	2.33	56.61	0.69	2.85	268
	25	2680	4.68	8.45	0.55	3.63	194
	26	2800	8.22	3.27	0.63	2.10	154
	27	1920	8.72	0.78	0.24	0.89	非晶質
	28	1930	8.32	0.95	0.28	1.11	23
	29	1980	8.08	0.78	0.27	1.56	非晶質

[0022] Measurement results, such as a coat degree of hardness which should be contributed to the oxidation resistance of the example of this invention shown in Table 2 in Table 1 and the example of a comparison, lubricity, and abrasion resistance, are shown. Oxidation resistance measured the increase of a weight around [by the oxidization at the time of holding in 900 degrees C among the atmospheric air] unit time. Lubricity was evaluated by measuring coefficient of friction with carbon steel. The degree of hardness computed Vickers hardness number from the indenter penetration depth in 1g load using the nano indenter. It is clear that the example's of this invention it is the result of excelling the example of a comparison also in any.

[0023]

[Table 3]

	試料番号	エンドミル寿命(m)	ドリル：スラスト/N	穴数	インサート寿命 hr
本発明例	1	65	125	760	1.54
	2	75	120	950	1.78
	3	48	127	578	1.22
	4	81	135	1016	1.88
	5	55	137	783	1.35
	6	55	116	852	1.41
	7	60	127	879	1.49
	8	51	132	853	1.45
	9	60	128	720	1.44
	10	69	120	823	1.60
	11	71	110	954	1.75
	12	75	108	1036	2.01
	13	67	121	979	1.86
	14	69	128	857	1.58
	15	61	129	891	1.60
比較例	16	2	185	21	0.11
	17	4	101	43	0.24
	18	27	188	257	0.75
	19	25	185	298	0.77
	20	31	184	358	0.81
	21	34	175	348	0.75
	22	29	116	211	0.45
	23	35	185	276	0.71
	24	14	190	88	0.33
	25	30	150	364	0.85
	26	36	140	484	1.03
	27	8	105	112	0.16
	28	12	101	153	0.31
	29	13	95	143	0.22

[0024] The tool life when carrying out end-mill cutting for the example shown in Table 1 under the following conditions is shown in Table 3.
Base composition 90WC-9.5Co-0.5 particle-size [Cr and WC] tool of 0.8 micrometers A six sheet blade, **-ed [of diameters / 8mm end-mill] material SKD11 (HRC63)
Cutting speed 100m / the amount of min slittings 8mm x 0.8mm feed per revolution 50 micrometers / blade dry type, and a wet The dry-type cutting life criterion was taken as the time of an end mill breaking. Also in any, the example of this invention is long lasting, and its effect of the multilayer structure which added Cr and oxygen in TiAlN system is clear.
[0025] The result at the time of carrying out a hole dawn manipulation on the conditions shown in Table 3 below in the example of this invention and the example of a comparison which were shown in Table 1 is written together. The thrust force is the result of measuring [initial 10 of cutting hole]. The number of holes when a drill breaks estimated the life.
Base composition 91, **-ed [5WC-8Co-0.5 Cr and WC particle-size] material SKD61 (HRC42) [of 0.8 micrometers] diameter of a drill 8mm cutting speed 80m / min feed per revolution 0.2mm / rev hole depth 32mm dry type and a wet one the example of a dry-type cutting this invention -- very -- the thrust force -- low -- a result -- being long lasting -- things -- being clear .
[0026] Next, the example of this invention and the example of a comparison were made as an experiment in the insert die of cemented carbides, and it cut by the following cutting conditionss. The result is written together to Table 3. These cutting conditionss are conditions from which a cutting speed becomes it is quick and important [the oxidation resistance of a tool] in face cutter cutting.
Base P30 grade cemented-carbide insertion SEE42TN (20 degree recess)
**-ed material SKD61 (HRC22)
Cutting speed It cuts deeply 400m / min. It sends 1mm. 0.1mm / blade dry type, and a wet The dry-type cutting life judging was taken as the cutting time until flank average wear becomes 0.4mm. In the example of this invention, the remarkable enhancement in a life was checked so that clearly from Table 3.
[0027]
[Effect] The enhancement in the coat adhesion by the thing for which not only making oxidation resistance improve but it gives lubricity without a wear-resistant degradation, and the reduction in stress is possible for TiAlCrON system multilayer which added Cr and oxygen to TiAlN system coat, and it can demonstrate the property which was excellent in high-speed dry-type cutting. Moreover, also in the intended use of hot forging etc., the effect is the same.

[Translation done.]

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最終頁に続く

(54)【発明の名称】　硬質皮膜被覆部材

(57)【要約】
【目的】硬質皮膜を被覆した硬質皮膜被覆部材において耐摩耗性、耐酸化性、潤滑性の特性を向上させることを目的とする。
【構成】硬質皮膜を被覆した硬質皮膜被覆部材において、該硬質皮膜は少なくともＡｌ、Ｔｉ、Ｃｒ、Ｎ、Ｏを含有する皮膜を１層乃至２層以上、被覆したことを特徴とする硬質皮膜被覆部材。

【特許請求の範囲】

【請求項１】硬質皮膜を被覆した硬質皮膜被覆部材において、該硬質皮膜は少なくともＡｌ、Ｔｉ、Ｃｒ、Ｎ、Ｏを含有する皮膜を１層乃至２層以上、被覆したことを特徴とする硬質皮膜被覆部材。

【請求項２】請求項１記載の硬質皮膜被覆部材において、該硬質皮膜を構成する層の組成は、

$(Al_aTi_bCr_c)(N_wO_{1-w})$、

但し、３０≦ａ≦７０、３０≦ｂ≦７０、０．５≦ｃ≦２０、

ａ＋ｂ＋ｃ＝１００、０．７≦ｗ≦０．９９、であることを特徴とする硬質皮膜被覆部材。

【請求項３】請求項１乃至２記載の硬質皮膜被覆部材において、該層数は３～１０００層であることを特徴とする硬質皮膜被覆部材。

【請求項４】請求項１乃至３記載の硬質皮膜被覆部材において、該層の膜厚は５ｎｍ～２０００ｎｍであることを特徴とする硬質皮膜被覆部材。

【請求項５】請求項１乃至３記載の硬質皮膜被覆部材において、硬質皮膜を酸素含有量の少ないＡ層と酸素含有量の多いＢ層で構成したことを特徴とする硬質皮膜被覆部材。

【請求項６】請求項５記載の硬質皮膜被覆部材において、該Ａ層の酸素含有量は１～１０原子%、該Ｂ層の酸素含有量は１０～３０原子%であることを特徴とする硬質皮膜被覆部材。

【請求項７】請求項５乃至６記載の硬質皮膜被覆部材において、該Ａ層及び／又はＢ層の一部もしくは全体に亘り、酸素含有量が傾斜組成を有することを特徴とする硬質皮膜被覆部材。

【請求項８】請求項１乃至７記載の硬質皮膜被覆部材において、該硬質皮膜の結晶構造がＮａＣｌ型であることを特徴とする硬質皮膜被覆部材。

【請求項９】請求項１乃至８記載の硬質皮膜被覆部材において、該硬質皮膜のＸ線回折における（２００）面の回折強度をＩ（２００）、同（１１１）面の回折強度をＩ（１１１）とした時、Ｉ（２００）／Ｉ（１１１）の値が１以上であることを特徴とする硬質皮膜被覆部材。

【請求項１０】請求項１乃至９記載の硬質皮膜被覆部材において、該硬質皮膜結晶形態が微細柱状結晶もしくは非晶質であることを特徴とする硬質皮膜被覆部材。

【請求項１１】請求項１乃至１０記載の硬質皮膜被覆部材において、該微細柱状結晶の粒径が、硬質皮膜と基体の界面から１０００ｎｍ～１５００ｎｍの位置において、２５０ｎｍ以下であることを特徴とする硬質皮膜被覆部材。

【請求項１２】請求項１乃至１１記載の硬質皮膜被覆部材において、該硬質皮膜に残留する圧縮応力が３．５ＧＰａ以下であることを特徴とする硬質皮膜被服部材。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は固体潤滑性に優れるとともに高い耐摩耗性、耐酸化性を有する耐摩耗性部材に関する。

【０００２】

【従来の技術】工具や金型及び機械部品において耐摩耗性、耐酸化性、潤滑性を付与するために各種硬質皮膜を被覆することが一般的になっている。代表的なＴｉＮ、ＴｉＣＮ皮膜は耐摩耗性は優れるものの、耐酸化性に問題を有する。また、特開昭６２－５６５６５などによって提案されたＴｉＡｌＮ系皮膜は耐摩耗性、耐酸化性に優れるものの潤滑性は低い。ＣｒＮ、ＣｒＣＮ系皮膜は潤滑性は優れるものの皮膜硬度が低く耐摩耗性に劣る。このように従来の皮膜は耐摩耗性、耐酸化性、潤滑性のいずれかが劣り、各用途において何らかの問題点を有する結果であるのが現状である。また潤滑性を付与するために硬質皮膜表面に潤滑性に優れるＭｏＳ系皮膜を被覆する提案が特開平５－２３９６１８などでなされてはいるが、密着性が悪く十分な結果を得るには至っていない。このように従来の皮膜では何らかの問題点があり、それを解決するためにＭｏＳ系以外において、特開平１１－１５６９９２に示されるようにＴｉＡｌＮ系皮膜の上層にＣｒＮ系皮膜を被覆する提案もなされているが、全体の膜厚はある程度の制限があるためＴｉＡｌＮ皮膜の膜厚が十分でなく耐摩耗性の点で満足されるものではない。

【０００３】

【発明が解決しようとする課題】本発明は耐摩耗性、耐酸化性、潤滑性のいずれをも劣化させることなく、これらの特性を向上させることを目的とする。

【０００４】

【課題を解決するための手段】上記の課題を解決するために本発明は、硬質皮膜を被覆した硬質皮膜被覆部材において、該硬質皮膜は少なくともＡｌ、Ｔｉ、Ｃｒ、Ｎ、Ｏを含有する皮膜を１層乃至２層以上、被覆する。また、本発明のより優れた実施態様として、硬質皮膜を構成する各層の組成は、$(Al_aTi_bCr_c)(N_wO_{1-w})$、但し、３０≦ａ≦７０、３０≦ｂ≦７０、０．５≦ｃ≦２０、ａ＋ｂ＋ｃ＝１００、０．７≦ｗ≦０．９９とすし、その層数は３～１０００層とする、各層の膜厚は５ｎｍ～２０００ｎｍとする、硬質皮膜は酸素含有量の少ないＡ層と酸素含有量の多いＢ層で構成したものとする、Ａ層の酸素含有量は１～１０原子%、Ｂ層の酸素含有量は１０～３０原子%とする、各層の一部もしくは各層全体に亘り酸素含有量は傾斜組成とする、硬質皮膜の結晶構造はＮａＣｌ型とする、硬質皮膜のＸ線回折における（２００）面の回折強度をＩ（２００）、同（１１１）面の回折強度をＩ（１１１）とした時、Ｉ（２００）／Ｉ（１１１）の値は１以上とする、硬質皮膜結晶形態は微細柱状結晶もしくは非晶質形

態とする、微細柱状結晶の粒径は硬質皮膜と基体の界面から１０００ｎｍ～１５００ｎｍの位置において２５０ｎｍ以下とする、硬質皮膜に残留する圧縮応力は３．５Ｇｐａ以下とする、などが挙げられる。本発明はＴｉとＡｌとＣｒとＮを必須成分とし、これに酸素を添加した硬質皮膜を採用するものである。当然ながら、ＴｉとＡｌが耐摩耗性成分として寄与し、Ｃｒが潤滑性を付与する成分として寄与するわけであるが、これだけでは十分でなく、これに酸素を添加することにより、いっそう耐酸化性、潤滑性を付与する結果となる。

【０００５】

【作用】ＴｉＡｌＮを基本とし、これにＣｒを添加する効果については、第一に特に切削工具において耐酸化性のさらなる向上が挙げられる。ＴｉＡｌＮの場合、酸化に伴い皮膜内部でＡｌが表面に拡散し、Ａｌの酸化物を形成することにより酸素の外部からの浸入を抑制し、耐酸化性が向上することは周知の事実である。しかしこの場合Ａｌの酸化物の直下には、非常にポーラスなＴｉの酸化物が形成されるため、特に切削工具などの衝撃が加わるような場合、Ａｌの酸化物は容易に脱落してしまい、その効果を継続して発揮することが難しい。金型等の場合でも同様である。Ｃｒの添加によりＡｌの酸化物直下に形成されるポーラスなＴｉの酸化物がＴｉＣｒの酸化物となり、この酸化物は非常に緻密な層を形成することが明らかとなった。従って、最表層に形成されるＡｌの酸化物は密着性が保たれ、結果耐酸化性が向上することになる。

【０００６】Ｃｒ添加の第二の効果はＣｒそのものが有する優れた潤滑性をＴｉＡｌＮ皮膜に付与することである。ＴｉＡｌＮの鋼との摩擦係数は０．７－０．８であるが、Ｃｒ添加に伴い０．３－０．６に改善される。この摩擦係数はＣｒの添加量に依存する。ただしＣｒの添加量が多すぎると皮膜硬度が低下し、耐摩耗性が悪くなるため、添加量には上限を設定することが好ましい。

【０００７】Ｃｒ添加のみでも上述のようにＴｉＡｌＮ系皮膜の潤滑性、耐酸化性を向上せしめる効果が確認されるが、Ｃｒのみでは十分ではなく、これに酸素を添加することにより、さらに一層の改善が認められる。酸素の添加効果については第一に耐酸化性の飛躍的向上、潤滑性の飛躍的向上が挙げられる。耐酸化性の飛躍的向上に対しては、皮膜内部に酸素を添加することに伴い、皮膜の結晶が微細化し皮膜そのものが緻密になると同時に結晶粒界も緻密になることにより、外部からの酸素の浸入に対する酸素拡散速度が著しく抑制されることが理由と考えられる。潤滑性に関しては、十分に解明されてはいないが、酸素添加により鋼との親和性が低下することが理由であると考えられる。

【０００８】酸素添加効果の第二は皮膜の残留圧縮応力が低下し、皮膜の密着性が向上することに起因する耐摩耗性の向上が挙げられる。特に重切削条件下や鍛造金型分野においては皮膜の密着性が極めて重要な要素となる。皮膜の微小剥離により摩耗が進行する傾向にあるとともに、大きな剥離が発生すると突発的寿命となる。ＴｉＡｌＣｒＮ系の皮膜のスクラッチテストによる剥離臨界荷重は６０－８０Ｎであるのに対し、これに酸素を添加することによりその強度は１００Ｎ以上に改善される。

【０００９】しかしながら、酸素の添加量増加に伴い、上述の耐酸化性、潤滑性、密着性向上に起因する耐摩耗性は向上するものの、一方皮膜硬度そのものが軟化してしまいアブレッシブ摩耗性が劣化する結果となる。従って耐酸化性、潤滑性に寄与するための最適組成層と、アブレッシブ磨耗に寄与する最適組成層の多層化することが重要で、より好ましく、多層化により双方の利点が複合化される結果となる。

【００１０】次に数値を限定した理由を説明する。Ａｌは３０原子%未満であると皮膜の耐酸化性が劣化し７０原子%を超えると皮膜中にｈｃｐ構造を有するＡｌＮが形成され皮膜強度が劣化するため好ましくない。Ｔｉは３０原子%未満であると皮膜の耐摩耗性が劣化し、７０原子%を超えると皮膜の耐酸化性が劣化するため好ましくない。Ｃｒは０．５原子%未満ではポーラスなＴｉ酸化物が形成され耐酸化性向上に寄与せず、２０原子%を超えると皮膜の硬度が軟化し耐摩耗性が劣化し好ましくない。酸素は窒素に対し１原子%未満では、耐酸化性、潤滑性、密着性の向上に寄与せず、３０原子%を超えると皮膜硬度の軟化を来すため好ましくない。

【００１１】多層における皮膜数は３層未満であると、それぞれの効果は発揮するものの、上述したようにどちらかの欠点が顕著となり複合効果が認められず、１０００層を超えると各層の膜厚が薄く同じく複合効果が認められなくなるとともに残留応力が増加し皮膜の密着性を損なう傾向があるため好ましくない。各層の膜厚も同様であり、５ｎｍ未満では各層の有する利点の効果が薄れ、２０００ｎｍを超えるとせいぜい３層程度の被覆しか現実性がなくなり好ましくない。

【００１２】低酸素含有層と高酸素含有層を多層にする目的は上述のように、低酸素皮膜は硬度低下が少なく耐アブレッシブ摩耗に寄与し、高酸素含有皮膜は皮膜硬度が低下する傾向にあるものの耐酸化、潤滑性に寄与する効果が大きく、これらを多層に被覆することにより双方の効果が複合化され好ましい結果となる。低酸素含有層において、酸素含有量が１原子%未満では、高酸素含有層との密着性が劣化し、１０原子%を超えると耐アブレッシブ摩耗性が劣化するため好ましくない。一方高酸素含有皮膜においては、酸素含有量が１０原子%未満では耐酸化性、潤滑性の向上に寄与が薄く、３０原子%を超えると著しく硬度が軟化し耐摩耗性を劣化させ好ましくない。

【００１３】この低酸素含有皮膜と高酸素含有皮膜は単

純な多層でも何ら問題はないが、各層において酸素含有量を傾斜化させ界面における酸素含有量の変化をできるだけ少なくするか、酸素含有量をサイン曲線のごとく連続化させることにより、各層の密着性がさらに向上する結果となる。

【００１４】結晶形態においては、ＮａＣｌ型が多数のすべり面を有するため、高温での皮膜硬度においてＨＶ３０００程度の上限値が存在し、これ以上の高硬度化は難しいものの、反面靭性に優れ、チッピングの発生、衝撃が加わった場合等における皮膜中のマイクロクラックの発生が少なく、安定した寿命が達成される。

【００１５】皮膜の結晶配向は被覆条件に依存し、比較的低エネルギーで成膜すると（２００）面に強く配向し、高エネルギーで成膜すると（１１１）面に配向する傾向を有する。低エネルギーで成膜した場合が皮膜の成膜速度は遅くなるが、皮膜密度が向上し、耐酸化性、耐磨耗性に優れた結果となることが確認された。従って（２００）面回折強度が（１１１）面回折強度より強い場合、さらに優れた耐酸化性、耐摩耗性を発揮し、より好ましいと考えられる。尚、潤滑性においては結晶配向は大きな影響を及ぼすものではなかった。

【００１６】皮膜の結晶粒径は破面ＳＥＭにて決定し、基体表面から１０００ｎｍ～１５００ｎｍの位置において基体に平行な線を設定し、これに交わる粒界の数より規定する。この場合皮膜結晶粒径が２５０ｎｍを超えて粗大化すると耐摩耗性、皮膜強度が劣化し好ましくない。ここで非晶質状というのは実際には非晶質ではないが、破面観察上明確な結晶粒界が観察されない場合をいう。このような場合特に耐酸化性の著しい向上が確認されている。

【００１７】皮膜に残留する圧縮応力は主に被覆条件に依存するが、３．５Ｇｐａを超えると密着性が劣化してしまうため、好ましくない。尚、本発明の皮膜はアークイオンプレーティング方式、スパッタリング方式、電子銃蒸発方式、プラズマアシストＣＶＤ方式のいずれであっても傾向は同様であり、またこれら各方式の組み合わせであってもよい。

【００１８】

【実施例】次に本発明の好ましい実施例を比較例とともに説明する。アークイオンプレーティング方式により本発明例と比較例の皮膜を作成した。ＡｌＴｉＣｒの組成は蒸発源であるカソードターゲットの金属組成を調整することにより調整した。酸素含有量は窒素と酸素の混合ガスを使用しその混合比を調整することにより調整するとともに、ガスの切り替えにより多層化した。結晶配向上は基本的には被覆条件で調整し、（２００）配向皮膜は被覆条件を基体に印可するバイアス電圧を７０Ｖ反応圧力を１Ｐａとし、（１１１）配向皮膜はバイアス電圧を２００Ｖ反応圧力を０．５Ｐａとすることにより作成した。Ｉ（２００）／Ｉ（１１１）比はこの他皮膜組成、酸素含有量にも多少依存するものである。

【００１９】

【表１】

	試料番号	A層	B層	層数
本発名例	1	50Al40Ti10Cr-5095N	50Al40Ti10Cr-25075N	20
	2	55Al35Ti10Cr-5095N	55Al35Ti10Cr-25075N	20
	3	35Al55Ti10Cr-5095N	35Al55Ti10Cr-25075N	20
	4	65Al32Ti3Cr-5095N	65Al32Ti3Cr-25075N	20
	5	33Al64Ti3Cr-5095N	33Al64Ti3Cr-25075N	20
	6	40Al35Ti25Cr-5095N	40Al35Ti25Cr-25075N	20
	7	50Al40Ti10Cr-2098N	50Al40Ti10Cr-25075N	20
	8	50Al40Ti10Cr-5095N	50Al40Ti10Cr-13087N	20
	9	50Al40Ti10Cr-5095N	50Al40Ti10Cr-25075N	4
	10	〃	〃	100
	11	〃	〃	500
	12	〃	〃	900
	13	50Al40Ti10Cr-(10-1-10)O(90-99-90)N傾斜	50Al40Ti10Cr-(10-25-10)O(90-75-90)N傾斜	20
	14	40Al35Ti25Cr-5095N	65Al32Ti3Cr-15085N	20
	15	33Al64Ti3Cr-7093N	40Al35Ti25Cr-15085N	20
比較例	16	TiN	-	1
	17	Ti-50N50C	-	1
	18	50Al50TiN	-	1
	19	TiN(500nm)	50Al50TiN(2500nm)	2
	20	65Al35TiN	-	1
	21	65Al35TiN	100nmMoS_2	2
	22	65Al35TiN(2000nm)	CrN(1000nm)	2
	23	50Al50Ti-70N30C	-	1
	24	TiN	50Al50TiN	20
	25	50Al40Ti10CrN	-	1
	26	50Al40Ti10Cr-5095N	-	1
	27	50Al40Ti10Cr-25075N	-	1
	28	50Al40Ti10Cr-5095N	50Al40Ti10Cr-50050N	20
	29	〃	〃	1500

【００２０】表１に本発明例と比較例の詳細を示す。尚皮膜の厚さは今回本発明例及び比較例においてすべて３０００ｎｍ～３２００ｎｍで統一した。

【００２１】

【表２】

	試料番号	硬度(HV)	I(200)/I(111)	酸化増量 mg/min	摩擦係数	応力 Gpa	粒径 nm
本発明例	1	2650	5.23	1.87	0.35	1.26	160
	2	2700	4.22	1.11	0.34	1.35	148
	3	2570	7.11	3.56	0.38	1.05	220
	4	2750	3.25	1.43	0.33	2.04	158
	5	2490	4.33	3.10	0.32	2.22	210
	6	2500	2.89	0.98	0.38	1.59	非晶質
	7	2670	2.56	1.98	0.39	2.46	201
	8	2710	2.77	2.31	0.40	2.41	175
	9	2630	6.14	1.78	0.35	1.02	210
	10	2690	4.26	1.55	0.34	1.96	143
	11	2750	3.44	1.43	0.33	2.31	121
	12	2800	2.49	1.40	0.33	2.93	98
	13	2660	5.03	1.79	0.32	1.76	158
	14	2600	4.34	1.23	0.38	1.88	125
	15	2590	3.89	1.49	0.33	1.97	128
比較例	16	2160	0.25	85.28	0.87	1.67	350
	17	2980	0.22	98.13	0.29	4.02	256
	18	2700	2.43	11.67	0.85	2.89	341
	19	2700	0.56	12.55	0.85	3.97	298
	20	2750	3.22	8.05	0.89	2.11	253
	21	2720	3.22	22.23	0.11	2.78	331
	22	2240	3.22	15.67	0.29	2.88	332
	23	3010	2.56	25.44	0.65	3.97	247
	24	2450	2.33	56.81	0.88	2.85	286
	25	2680	4.88	6.45	0.55	3.63	194
	26	2600	6.22	3.27	0.53	2.10	154
	27	1920	8.72	0.78	0.24	0.89	非晶質
	28	1930	6.32	0.98	0.28	1.11	23
	29	1980	6.08	0.76	0.27	1.56	非晶質

【００２２】表２に表１で示した本発明例及び比較例の耐酸化性、潤滑性及び耐摩耗性に寄与すべき皮膜硬度等の測定結果を示す。耐酸化性は大気中９００℃において保持した場合の酸化による単位時間あたりの重量増を測定した。潤滑性は炭素鋼との摩擦係数を測定することにより評価した。硬度はナノインデンターを用い１ｇ荷重における圧子侵入深さよりビッカース硬さを算出した。本発明例はいずれにおいても比較例より優れる結果であることが明らかである。

【００２３】

【表３】

	試料番号	ｴﾝﾄﾞﾐﾙ寿命(m)	ﾄﾞﾘﾙ：ｽﾗｽﾄ /N	穴数	ｲﾝｻｰﾄ寿命 hr
本発明例	1	65	125	760	1.54
	2	75	120	950	1.78
	3	48	127	578	1.22
	4	81	135	1016	1.88
	5	55	137	783	1.35
	6	55	116	852	1.41
	7	60	127	679	1.49
	8	51	132	653	1.45
	9	60	128	720	1.44
	10	69	120	823	1.60
	11	71	110	954	1.75
	12	75	108	1036	2.01
	13	87	121	979	1.86
	14	63	128	857	1.56
	15	61	129	891	1.60
比較例	16	2	195	21	0.11
	17	4	101	43	0.24
	18	27	189	257	0.75
	19	25	185	298	0.77
	20	31	186	358	0.81
	21	34	175	348	0.75
	22	29	115	211	0.45
	23	35	165	278	0.71
	24	14	190	86	0.33
	25	30	150	364	0.85
	26	36	140	484	1.03
	27	8	105	112	0.16
	28	12	101	153	0.31
	29	13	95	143	0.22

【００２４】表３に表１に示した例を以下の条件下でエンドミル切削を実施した時の工具寿命を示す。

基体組成　９０WC－９．５Co－０．５Cr、WC粒径０．８μm
工具　６枚刃、径８mmエンドミル
被削材　ＳＫＤ１１（ＨＲＣ６３）
切削速度　１００m／ｍｉｎ
切り込み量　８mm　ｘ　０．８mm
送り量　５０μm／刃
乾式・湿式　乾式切削
寿命判定基準はエンドミルが折れる時点とした。いずれにおいても本発明例は長寿命でありＴｉＡｌＮ系にＣｒと酸素を添加した多層構造の効果が明らかである。

【００２５】表３に表１に示した本発明例及び比較例において以下に示す条件で穴明け加工した場合の結果を併記する。スラスト力は切削初期１０穴目において測定した結果である。寿命はドリルが折損した時の穴数で評価した。

基体組成　９１、５WC－８Co－０．５Cr、WC粒径０．８μm
被削材　ＳＫＤ６１（ＨＲＣ４２）
ドリル径　８mm
切削速度　８０m／ｍｉｎ
送り量　０．２mm／ｒｅｖ
穴深さ　３２mm
乾式・湿式　乾式切削
本発明例は極めてスラスト力が低く、結果長寿命であることが明らかである。

【００２６】次に超硬インサートにて本発明例及び比較例を試作し、以下の切削条件で切削を実施した。その結果を表３に併記する。本切削条件は正面フライス切削では切削速度が速く工具の耐酸化性が重要となる条件である。

基体　Ｐ３０グレード超硬合金
インサート　ＳＥＥ４２ＴＮ（２０°逃げ）
被削材　ＳＫＤ６１（ＨＲＣ２２）
切削速度　４００m／ｍｉｎ
切り込み　１mm
送り　０．１mm／刃
乾式・湿式　乾式切削
寿命判定は逃げ面平均摩耗が０．４mmに達するまでの切削時間とした。表３から明らかなように、本発明例では著しい寿命向上が確認された。

【００２７】

【効果】ＴｉＡｌＮ系皮膜にＣｒと酸素を添加したＴｉＡｌＣｒＯＮ系多層膜は耐酸化性を向上せしめるのみでなく、耐摩耗性の劣化なく潤滑性を付与すること、並びに、低応力化による皮膜密着性の向上が可能であり高速乾式切削において優れた特性を発揮することが可能である。また熱間鍛造の用途等においてもその効果は同様である。

フロントページの続き

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